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मानक

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“Step Out From the Old to the New”

IS 12336 (1988): Method for determination of diazinon residues in food commodities [FAD 1: Pesticides and Pesticides Residue Analysis]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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*Indian Standard***METHOD FOR DETERMINATION OF DIAZINON
RESIDUES IN FOOD COMMODITIES****1. Scope**

1.1 This standard prescribes gas chromatographic method (GLC) for the determination of diazinon [00 - diethyl 0 - (2-isopropyl-6-methyl-4-pyrimidinyl) phosphorothionate or diethyl-2-isopropyl-6-methyl-4-pyrimidinyl phosphorothionate] residues in food commodities.

1.1.1 Though no set procedure for thin layer chromatography (TLC) is being prescribed, standardized TLC may be followed, if necessary, for the purpose of identification and confirmation of diazinon residues.

2. **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water [see IS : 1070 - 1977 Specification for water for general laboratory use (*second revision*)] shall be employed in tests.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. Sampling

3.1 The representative samples for the purpose of estimating diazinon residues in food commodities shall be drawn in accordance with IS : 11380 (Part 1) - 1985 'Method of sampling for the determination of pesticide residues : Part 1 Agricultural and food commodities'.

4. Method**4.1 Apparatus**

4.1.1 *Waring blender or equivalent* — fire and explosion proof.

4.1.2 *Kuderna Danish concentrator or equivalent*

4.1.3 *Chromatographic column* — 200 cm long, 18 mm ID glass column with grease free stopcock.

4.1.4 *Filtering equipment*

4.1.5 *Air blower*

4.1.6 *Rotary vacuum evaporator*

4.1.7 *Mechanical shaker*

4.1.8 *Water pump for suction*

4.2 Reagents

4.2.1 *n-Hexane* — analytical reagent grade, glass redistilled.

4.2.2 *Benzene* — analytical reagent grade, glass redistilled.

4.2.3 *Methyl alcohol* — analytical reagent grade, glass redistilled.

4.2.4 *Methylene chloride* — analytical reagent grade, glass redistilled.

4.2.5 *Cyclohexane* — analytical reagent grade, glass redistilled.

4.2.6 *Sodium chloride* — analytical reagent grade.

4.2.7 *Sodium sulphate* — anhydrous.

4.2.8 *Acetonitrile* — analytical reagent grade, glass redistilled.

4.2.9 *Ethyl acetate* — chromatographic grade, glass redistilled.

4.2.10 *Activated alumina* — Basic-Grade I. Prepare activity Grade III by mixing 100 g with 6 ml water. Mix thoroughly and allow to stand overnight in a sealed bottle.

4.2.11 *Activated charcoal* — Nuchar SG — extra or equivalent.

4.3 Extraction

4.3.1 *Fruits, vegetables and root crops* — Transfer 50 g of the finely chopped material into a blender jar. Add 400 ml methanol and blend at high speed for 2 minutes. Transfer the macerate to a 2-litre wide-neck bottle and shake mechanically for 2 to 4 hours. Filter the extract using gentle

Adopted 30 March 1988

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suction. Wash residue in the funnel with two 50-ml portions of methanol. Transfer the filtrate to 1-litre graduated cylinder and record the volume. Transfer half of the solution corresponding to 100 g sample to 1-litre separating funnel, add 400 ml water and 10 ml saturated sodium chloride solution and shake vigorously with two 100-ml portions of methylene chloride. Separate methylene chloride extracts, combine, dry with anhydrous sodium sulphate and evaporate to dryness in a rotary vacuum evaporator (Extract A).

4.3.2 Oils and *fats* — Transfer 20-25 g material into a 400-ml separating funnel and shake with two 50-ml portions of acetonitrile. Combine the acetonitrile extracts and wash 50 ml n-hexane in a 250-ml separating funnel. Evaporate acetonitrile extract to dryness (Extract B).

4.3.3 Corn, *foodgrains* and fodder *material* — Transfer 250 g of sample to a blender jar. Add 500 ml ethyl acetate and blend for five minutes at high speed. Add 20 g Nuchar SG, extra charcoal (see 4.2.1 1) and blend the mixture at a low speed for two minutes. Filter the contents by gravity and make up to 100 ml. Transfer 10 ml extract to a 15 ml graduated centrifuge tube and concentrate to 5 ml on a warm water bath, with a gentle stream of dry air (Extract C).

4.4 Clean Up

4.4.1 Fill chromatographic column with n-hexane and 25 g alumina grade (see 4.2.10) to obtain a column height of at least 10 cm. Dissolve extract A or B in 2 ml benzene and transfer to the column. Wash the column with hexane twice and collect elute in 250 ml flask when the last portion of hexane has just entered adsorbant. Continue elution with 100 ml 5 percent ethyl ether in hexane at about 5 ml per minute. Evaporate the elute to dryness. Add known volume of hexane-ethyl ether mixture to the residue so that 5 μ l injection will produce approximately half-scale recorder deflection.

5. Determination — Gas Chromatographic Method

5.1 *Principle* — The diazinon residue extracted from samples is measured gas chromatographically using a NPD detector or flame photometric detector and a 526 nm interference filter (phosphorus specific). The content of diazinon is determined by comparing the response with a reference standard of diazinon.

5.1.1 *Gas chromatograph* — A gas chromatograph equipped with NPD detector or flame photometric detector is operated under the following suggested parameters. These parameters may be varied according to the available facilities provided standardization is done:

Column --- Glass, 61-cm long and 4-mm ID packed with 2 percent (w/w) diethylene glycol succinate on Gas Chrom. Q (100-120 mesh)

Column oven temperature	: 165°C
Injection port temperature	: 185°C
Detector temperature	: 150°C
Carrier gas	: Nitrogen
Carrier gas flow rate	: 100 ml/minute
Other (combustion) gases and their flow rate	: Oxygen — 50 ml/minute, or Air — 50 ml/minute, or Hydrogen — 200 ml/minute.

5.2 *Procedure* -- Inject 5 μ l of the extract obtained directly in 4.3.3 or 4.4.1 after clean up, as the case may be, into the column of the gas-chromatograph. Identify the peak for diazinon by its retention time and measure the peak height or area.

For the reference standard, prepare ethyl acetate solutions containing 0.01 to 10 μ g/ml of diazinon. This procedure is sensitive to 0.01 μ g/g.

5.3 Calculation

$$\text{Diazinon residue, } \mu\text{g/g (ppm)} = \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

A_1 = peak height/area of the sample;

V_2 = volume, in μ l, of standard diazinon injected;

V_3 = total volume, in ml, of sample solution;

C = concentration, μ g/g, of standard diazinon solution;

f = recovery factor = $\frac{100}{\text{percent mean recovery}}$

A_2 = peak height/area of the diazinon standard;

V_1 = volume, in μ l, of sample injected; and

M = mass, in g, of sample taken for analysis.

Note — Percent mean recovery is determined by taking untreated control sample to which known amount of diazinon is added and analysed as described above.

E X P L A N A T O R Y N O T E

Diazinon formulations are extensively used in agriculture for the control of a wide range of insect pests. This standard will enable the food, health authorities and others engaged in the fields to follow uniform test procedure for the estimation of diazinon residues in various food commodities.

In the preparation of this standard, due consideration has been given to the limits of diazinon residues laid under the provisions of *Prevention of food Adulteration Rules, 1965* and the test method is sensitive to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 'Rules for rounding off numerical values (*revised*)'.

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